The Patent Office

EP00/03724

INVESTOR IN PEOPLE

4

EPO - DG 1

20. 04. 2000

(55)

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed

Dated 17 February 2000

[redacted]ts Form 1/77

The Patent Office

Patents Act 1977
(Rule 16)

THE PATENT OFFICE
F
30 APR 1999
RECEIVED BY HAND

04MAY99 E444237-3 D02898
P01/7700 0.00 [redacted]

Cardiff Road
Newport
Gwent NP9 1RH

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

1. Your reference — C3913(C)

2. Patent application number *(The Patent Office will fill in this part)* — 30 APR 1999 — 9910101.6

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

UNILEVER PLC

UNILEVER HOUSE, BLACKFRIARS
LONDON, EC4P 4BQ

Patents ADP number *(if you know it)* 1628002

If the applicant is a corporate body, give the country/state of its incorporation — **UNITED KINGDOM**

4. Title of the invention — **CONCENTRATED PERFUME COMPOSITIONS AND MANUFACTURE OF A FABRIC SOFTENING COMPOSITION THEREFROM**

5. Name of your agent *(if you have one)* — **ELLIOTT Peter William**

"Address for Service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

PATENT DEPARTMENT, UNILEVER PLC
COLWORTH HOUSE, SHARNBROOK
BEDFORD, MK44 1LQ

Patents ADP number *(if you know it)* 5995477002

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day/month/year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

YES

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form	
Description	16
Claim(s)	3
Abstract	1
Drawing(s)	-

10. If you are also filing any of the following, state how may against each item.

Priority Documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	1
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature(s) Date: **30 April 1999**

Sandra Jane EDWARDS, Authorised Signatory

12. Name and daytime telephone number of person to contact in the United Kingdom — **Trudi Clark, (01234) 22 2360**

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
b) Write your answers in capital letters using black ink or you may type them.
c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
d) If you have answered 'Yes' Patents Form 7/77 will need to be filed.
e) Once you have filled in the form you must remember to sign and date it.
f) For details of the fee and ways to pay please contact the Patent Office.

Concentrated Perfume Compositions and Manufacture of a Fabric Softening Composition Therefrom

Technical Field

The present invention relates to concentrated perfume compositions. The invention further relates to a method of manufacturing a fabric softening composition from the concentrated perfume composition, and, to a fabric softening composition so produced.

Background and Prior Art

In the commercial manufacture of fabric softening compositions the accurate dosing of minor ingredients e.g. dyes, perfumes etc. is often problematic because of the low levels of these ingredients required in the compositions. Also when different compositions are produced in batches this requires, generally, that a composition has to be produced for each batch as the exact levels of minor ingredients, particularly dye and perfume, must be added individually which increases the possibility of inaccurate dosing.

The present invention seeks to address the above problems and to provide a stable concentrated composition which comprises at least two minor ingredients found in fabric softening compositions and an improved method of commercially producing fabric softening compositions.

Definition of the Invention

Accordingly the present invention provides a liquid composition comprising;

(a) 15 - 95 wt% lipophilic perfume,
(b) 0.05 - 5 wt% water-soluble dye, and
(c) 4 - 50 wt% of a cationic and/or nonionic stabilising agent

wherein the composition comprises between 0.1-20 wt% water and the cationic stabilising agent has an Lα to Lβ transition temperature of 45°C or below for a 5% wt dispersion of the stabilising agent in water.

The invention also provides a method of preparing a fabric softening composition comprising the steps;

(i) preparing an aqueous based composition comprising a cationic and/or nonionic fabric softening agent, and
(ii) adding to (i) a composition of the invention to produce the fabric softening composition.

Furthermore the invention also provides a fabric softening composition produced by the method above.

The invention provides a concentrated perfume composition that also contains dye in a much higher concentration than would typically be found in a fabric softening composition. This provides a stable pre-mix of minor ingredients, perfume and dye, which can be prepared at suitable ratios for direct dosage into a base composition. Excellent accuracy of the dosage of

minor ingredients into a base composition is achieved and simplifies automated preparation of fabric softening compositions.

Furthermore the concentrated perfume and dye compositions allow accurate dosing of these minor ingredients to a base composition at a late stage of the manufacture of a fabric softening composition. This in turn allows a wide range of compositions to be prepared from a pre-compounded base composition so providing easier and more versatile manufacture. Thus changing between variant formulations is simplified as only a small part of the automated production apparatus requires cleaning when the 'minors' are changed in the composition (as a single 'minor' composition can be used in the process). This provides reduced aqueous effluent, saves production time and provides increased production flexibility.

In particular the invention provides the preparation of a concentrated composition of perfume and dye, wherein the perfume and dye are of different lipophilic and lipophobic characters.

Detailed Description of the Invention

The composition of the invention is preferably an isotropic liquid, more preferably a microemulsion, and especially a water-in-oil microemulsion.

It is preferred that if the composition is an isotropic liquid it does not contain liquid crystalline phases.

Where the compositions are not clear, they should be stable to storage at 20°C for several days. Whist some degree of cloudiness can be tolerated in the compositions, it is preferred that they are isotropic liquids. Such isotropic liquids may have included therein minor amounts of materials that are not isotropic provided that the stability of the composition is not adversley affected.

The perfume used in the invention is lipophilic in nature. By a lipophilic perfume is meant that the perfume has a solubility in water (i.e. it dissolves) of 1g or less in 100 ml of water at 20°C. Preferably solubility in water is 0.5g or less, preferably 0.3g or less. Such perfumes may be referred to as water-insoluble perfumes.

The perfume may be any conventional perfume used in fabric softening compositions. The perfume will thus preferably be comparable with the types fabric softening actives typically found in fabric softening compositions, although, not many commercially available perfumes will not be compatible. Also the perfume will generally be polar in nature.

When the composition is a water-in-oil microemulsion the perfume will, because of its lipophilic nature, form the predominant part of the oil phase. It is preferred if the perfume comprises 60% by weight or more, preferably 70% by weight or more, of the oil phase when the composition is a water-in-oil microemulsion.

Perfumes contain a number of ingredients which may be natural products or extracts such as essential oils, absolutes,

resinoids, resins etc. and synthetic perfume components such as hydrocarbons, alcohols, aldehydes, ketones ethers, acids, esters, acetals, ketals, nitriles, phenols, etc. including saturated and unsaturated compounds, aliphatic, alicyclic, heterocyclic and aromatic compounds. Examples of such perfume components are to be found in "Perfume and Flavour Chemicals" by Steffen Arctander (Library of Congress catalogue card no. 75-91398).

Any lipophilic perfume which is compatible with nonionic and/or cationic compounds may be used in the composition.

The compositions contain 15 - 95 wt% of lipophilic perfume, preferably 20 - 90 wt%, more preferably 25 - 85 wt%, such as 40 - 85 wt%, e.g. 45 - 80 wt%.

More than one lipophilic perfume may be used in the compositions of the invention.

The dye is an at least a sparingly water-soluble dye and may be any such dye conventionally used in softening and cleaning products. It is especially preferred that the dye has a solubility in water of equal to, or greater than, 2g in 100 ml of water at 20°C, preferably 5g or more.

The dye may be an acid-dye or other suitable type of dye.

The dye is present in an amount of 0.05 - 5 wt%, preferably 0.1 - 2 wt%, more preferably 0.2 - 1 wt%, eg 0.25 - 0.7 wt%.

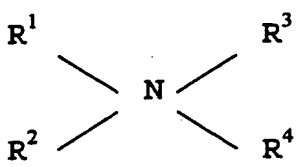
The weight ratio of perfume to dye is preferably within the range 200:1 to 5:1, more preferably 150:1 to 10:1, e.g. 100:1 to 15:1, such as 80:1 to 20:1. Depending upon the amount of dye required the ratio could be towards the lower end of the ratio e.g. 40:1 to 25:1. However if only a small amount of dye is required the ratio may be higher e.g. 900:1 to 250:1.

Where the stabilising agent comprises a cationic stabilising agent this has an Lα to Lβ transition temperature of 45°C or less for a 5 wt% dispersion of the agent in water. Cationic surfactants are the preferred stabilisng agents. This Lα to Lβ transition can be measured by DSC as defined in "Handbook of Lipid Bilayers", D Marsh, CRC Press, Boca Raton, Florida, 1990 (pages 137 and 337).

Any cationic stabiliser meeting the above transition temperature requirement, or, a nonionic stabilising agent, or mixtures thereof, may be used according to the invention.

The cationic stabilising agent used in the invention is preferably a cationic surfactant, more preferably one of the quaternary ammonium compounds of formulae (A) below or of (i) or (ii) further below. Compounds of these latter formulae are only stabilising agents as referred to herein if they meet the above transition temperature requirement.

(A)



wherein R^1 and R^2 are independently C_1-C_6 alkyl, alkenyl, substituted alkyl or akenyl groups or hydroxyalkyl groups and R^3 and R^4 are independently C_8-C_{28} alkylalkenyl substituted alkyl or alkenyl groups or hydroxalkyl groups. Preferably R^1 and R^2 are independently C_1-C_2 groups and R^3 and R^4 are independently C_{12}-C_{22} groups, X^- is a compatible anion eg Cl^-, $MESO_4^-$, Br^-, I^- acetate etc.

A cationic surfactant stabiliser according to formula (A) is ditallowyl dimethyl ammonium chloride.

A cationic stabiliser according to formula (i) below is dioleyl ester of methyl triethanol ammonium methosulphate wherein one R1 is methyl and one is hydroxyethyl, both n are 2, both T are O-C=O, and both R2 are tallow.

For cationic surfactants covered by the general formulae (i) and (ii) below only those meeting the transition temperature requirement above are cationic stabilisers as defined herein.

It is preferred that the stabilising agent is compatible with conventional fabric softening agents, and in particular with the fabric softening agents described hereinbelow. Mixtures of cationic stabilising agents may also be included.

Nonionic stabilising agents may also be used in addition to, or instead of, the cationic stabilising agent. Preferably the nonionic stabilising agent is a nonionic surfactant. Suitable types of nonionic surfactants include alcohol alkoxylates especially ethoxylates, preferably C8-C20 alkyl esters alkoxylated with an average of 1 to 10 alkoxylate units,

preferably 1 to 7 alkoxylate units. The ethoxylates, especially secondary alcohol ethoxylates, are particularly preferred.

The total amount of cationic and/or nonionic stabilising agent in the compositions is 4 wt% - 50 wt%, preferably 10-30 wt%, more preferably 15-25 wt%. Where a mixture of cationic and nonionic stabilisers are used the weight ratio of cationic:nonionic is preferably in the range 99:1 to 50:50, more preferably 99:1 to 60:40.

Where the stabilising agent comprises a nonionic surfactant then it is preferred that it has an HLB or 8 or below, preferably 7 or below.

The weight ratio of perfume to stabilising agent is preferably within the range 10:1 to 1:1, more preferably 8:1 to 1:1, e.g. 5:1 to 1:1, e.g. 3:1 to 1:1.

The compositions may also comprise minor amounts water-miscible solvents, typically in amounts of up to 10wt% of the composition, preferably up to 7.5 wt%.

The water-miscible solvent may be solvent having a C1-C6 alkyl chain such as ethanol or isopropanol. The solvent may be present in the compositions either through direct or may be added as being present in the stabilising agent or other component of the composition.

The compositions may also include low amounts (up to 5% by weight) of 'minor' ingredients typically found in fabric

softening compositions, provided, the stability of the composition is not affected. 'Minor' ingredients that may be included include fatty acids, non-aqueous solvents, fluorescers, hydrotropes, antifoaming agents, anti-redeposition agents, enzymes, optical brightening agents, opacifiers, anti-shrinking agents, anti-wrinkle agents, anti-spotting agents, germicides, fungicides, anti-oxidants, UV absorbers (sunscreens), sequestrants, preservative, chlorine scavengers, pH buffering agents, dye fixatives, anti-corrosion agents, drape imparting agents, and antistatic agents.

The total amount of the components in the composition always adds up to 100% by weight to include the essential components of the composition and minor ingredients, water, etc.

The compositions comprise 0.1-20 wt% water, preferably 0.1-15 wt%, more preferably 0.1-10 wt%.

Any suitable method of preparing the compositions of the invention may be used. For example the perfume, stabilising agent and any other oil soluble ingredients are mixed together to form a clear liquid. Gentle heating may be necessary at this point to produce the clear liquid (typically heating at 25-45°C). Water-soluble ingredients including the dye (and other 'minor' ingredients such as preservative) are dissolved in the required amount of water. The water-soluble aqueous portion is added to the perfume containing mixture, in aliquots if required, with stirring to produce the composition.

A further aspect of the present invention provides a method of producing a fabric softening composition by adding a

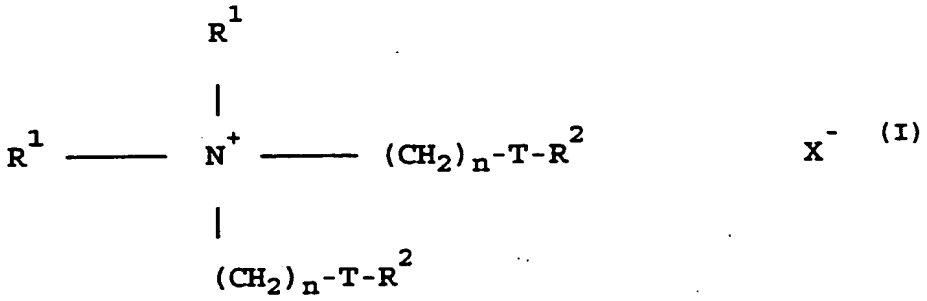
composition of the invention to a base composition comprising a cationic and/or nonionic fabric softening agent. Preferably the base composition is aqueous. The addition may be made in any suitable manner.

The composition is added at a suitable % by weight to give the required amount of perfume and dye etc. in the resultant fabric softening composition. The addition amount, and mixing, can be easily checked by measuring the colour. The fabric softener composition produced by the above method contains cationic and/or nonionic fabric softening agents.

Types of cationic fabric softeners that may be used in the fabric softening compositions produced from the compositions of the invention include substantially water-insoluble quaternary ammonium materials, for example, a compound having two C_{12}-C_{22} alkyl or alkenyl groups connected to a quaternary ammonium head group or a compound comprising a single long chain with an average chain length equal to or greater than C_{20}. Preferably these are connected to the quaternary ammonium head group via at least one ester link

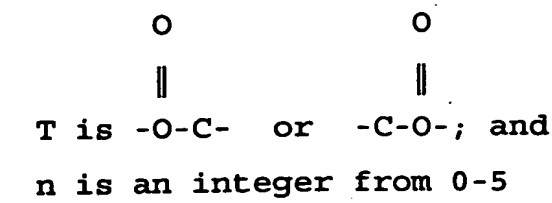
More preferably the invention is useful for quaternary ammonium material comprising a compound having two long chain alkyl or alkenyl chains with an average chain length equal to or greater than C_{14}. Even more preferably each chain has an average chain length equal to or greater than C_{16}. Most preferably at least 50% of each long chain alkyl or alkenyl group has a chain length of C_{18}.

It is preferred if the long chain alkenyl or alkenyl groups are predominantly linear. The especially preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula (I) :



(I)

wherein each R^1 group is independently selected from C_{1-4} alkyl, hydroxyalkyl or C_{2-4} alkenyl groups; and wherein each R^2 group is independently selected from C_{8-28} alkyl or alkenyl groups; X^- is any suitable counterion, for instance a halide, acetate or lower alkosulphate ion, such as chloride or methosulphate.

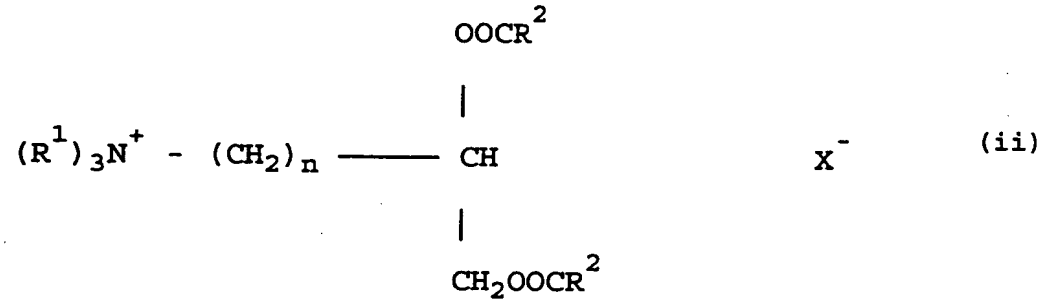


T is -O-C- or -C-O-; and

n is an integer from 0-5

Di (tallowyloxyethyl) dimethyl ammonium chloride, available from Hoechst, is especially preferred, also Di(hardened tallowyloxyethyl)dimethyl ammonium chloride, ex Hoechst).

A second preferred type of quaternary ammonium material can be represented by the formula (ii):



wherein R^1, n R^2 and X^- are as defined above.

It is advantageous for environmental reasons if the quaternary ammonium material is biologically degradable.

Preferred materials of this class such as 1,2 bis[hardened tallowoyloxy]-3- trimethylammonium propane chloride and their method of preparation are, for example, described in US 4 137 180 (Lever Brothers). Preferably these materials comprise small amounts of the corresponding monoester as described in US 4 137 180 for example 1-hardened tallow-oyloxy-2-hydroxy -3-trimethylammonium propane chloride.

The fabric softeners used in the compositions produced from the compositions of the invention are not required to meet the Lα to Lβ transition temperature referred to above.

Substantially water-insoluble fabric softening compounds are defined as fabric softening compounds having a solubility of less than 1 x 10-3 wt % in demineralised water at 20oC. Preferably the fabric softening compounds have a solubility of less than 1 x 10-4 wt%, more preferably less than 1 x 10-8 to 1 x 10-6 wt%.

The fabric softening compositions typically contain 1-8wt% of the fabric softening compound, and are known as dilute

compositions. They may contain higher amounts , such as 8-50% softening compounds in which case they are known as concentrates.

Nonionic fabric softeners may also be used, for example sorbitan esters and glycerol monostearate.

Further ingredients, typically minor ingredients, may be added to the fabric softeners so produced.

The present invention will be exemplified by way of the following examples. Further examples within the scope of the invention will be apparent to the skilled person.

Example 1; concentrated perfume composition

The composition below was prepared by mixing the perfume and Tetranyl AO-1 to form a clear liquid. Gentle warming was used. The dye and water were mixed to form a solution and this was added in aliquots to the perfume solution to produce the composition.

	grams of active ingredient	% by weight
Perfume*1	34.2	70.2
AO-1*2	10.0	20.5
Patent blue dye (10% active)	2.0	0.42
Isopropanol	5.0	5.0
Water		3.7

*1 - Softline 2000 perfume (ex Gividan Roure)

*2 - cationic surfactant, dioleyl ester of methyl triethanol ammonium methosulphate (Ex Kao).

Example 2; concentrated perfume composition

The composition below was prepared according to the method of example 1.

	grams of active ingredient	% by weight
Perfume*1	34.2	69.5
Prapagen 3445*3	10.0	20.2
Patent blue dye (10% active)	2.0	0.4
Isopropanol	4.04	4.04
Water		6.32

*3 - cationic surfactant ditallowyl dimethyl quaternary ammonium chloride (ex Clariant).

Example 3; concentrated perfume composition

The composition below was prepared according to the method of example 1.

	grams of active ingredient	% by weight
Perfume*4	34.2	70.2
AO-1 *2	10.0	20.5
Patent blue dye (10% active)	2.0	0.4
Isopropanol	5.0	5.0
Water	3.7	3.7

*4 - Horizon 2000 (ex IFF).

Examples 1 to 3 were all stable, isotropic water in oil microemulsions.

Examples 4; use of example 1 to prepare a fabric softening composition

A fabric softening base composition comprising 94.5 parts water and 5 parts dihardened tallow dimethyl ammonium chloride was prepared. To this 0.5 parts of example 1 was added and the composition stirred until homogeneous (to provide approximately 0.37% perfume, 0.11% AO-1 and 0.002% dye). A stable fabric softening composition was produced that showed the same physical characteristics as the comparative example below.

A comparative example was produced by the conventional method of mixing together the following ingredients:

	grams of active ingredient	% by weight
Dihardened tallow dimethyl ammonium chloride	5.0	5.0
Perfume*1	0.3	0.3
Patent blue dye (1% active)	0.002	0.002
Water	92.7	to 100%

The fabric softening composition comparative example and the fabric softening composition produced from example 1 both exhibited good stability on storage at room temperature

Claims -

1. A liquid composition comprising;
   (a) 15 - 95 wt% lipophilic perfume,
   (b) 0.05 - 5 wt% water-soluble dye, and
   (c) 4 - 50 wt% of a cationic and/or nonionic stabilising agent
   wherein the composition comprises between 0.1 to 20 wt% water and the cationic stabilising agent has an Lα to Lβ transition temperature of 45°C or less for a 5 wt% dispersion of the stabilising agent in water

2. A composition according to claim 1 wherein the composition is an isotropic liquid.

3. A composition according to claim 2 wherein the isotropic liquid is a water in oil microemulsion.

4. A composition according to any one of the preceding claims comprising 40-85 wt% perfume.

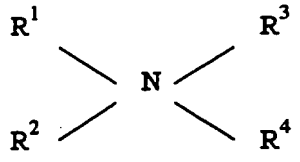
5. A composition according to any one of the preceding claims wherein the perfume has a solubility in water of equal to, or less than, 0.5g in 100 ml of water at 20°C.

6. A composition according to any one of the preceding claims comprising 0.2 wt% to 1 wt% dye.

7. A composition according to any one of the preceding claims wherein he dye has a solubility in water of equal to or greater than, 5g in 100 ml of water at 20°C.

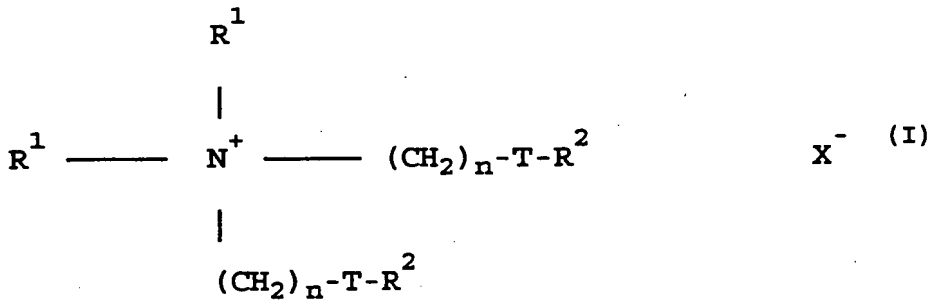
8. A composition according to any one of the preceding claims comprising 10 wt% - 30 wt% cationic surfactant as the stabilising agent.

9. A composition according to any one of the preceding claims wherein the cationic stabilising agent is a compound of general formula (A)

(A) 

wherein R^1 and R^2 are independently C_1-C_6 alkyl, alkenyl, substituted alkyl or akenyl groups or hydroxyalkyl groups and R^3 and R^4 are independently C_8-C_{28} alkylalkenyl substituted alkyl or alkenyl groups or hydroxalkyl groups

or, a compound of general formula (I)



wherein each R^1 group is independently selected from C_{1-4} alkyl, hydroxyalkyl or C_{2-4} alkenyl groups; and wherein each R^2 group is independently selected from C_{8-28} alkyl or alkenyl groups; X^- is chloride or methosulphate.

T is -O-C- or -C-O-; and

n is an integer from 0-5

10. A composition according to any one of the preceding claims wherein the ratio of perfume to dye is within the range 200:1 to 10:1, preferably 100:1 to 15:1.

11. A composition according to any one of the preceding claims wherein the ratio of perfume to cationic and/or nonionic stabilising agent is 10:1 to 1:1, preferably 5:1 to 1:1.

12. A composition according to any one of the preceding claims comprising 0.1- 10 wt% water.

13. A method of preparing a fabric softening composition comprising the steps;

(i) preparing a base composition comprising a cationic and/or nonionic fabric softening agent, and

(ii) adding to (i) a composition according to any one of the preceding claims,

to produce the fabric softening composition.

14. A fabric softening composition produced by the method of claim 13.

Abstract

The invention provides liquid compositions comprising:

(a) 15 - 95 wt% lipophilic perfume,
(b) 0.05 - 5 wt% water-soluble dye, and
(c) 4 - 50 wt% of a cationic and/or nonionic stabilising agent

wherein the composition comprises between 0.1 to 20 wt% water and the cationic stabilising agent has an Lα to Lβ transition temperature of 45°C or less for a 5 wt% dispersion of the stabilising agent in water

These concentrated perfume and dye compositions find particular application in fabric softening compositions.

Also provided is a method of preparing a fabric conditioning composition by preparing a base composition comprising a cationic and/or nonionic fabric softening agent, and adding thereto, a composition of the invention. The fabric conditioning compositions thus produced are also provided. Simplified automated manufacture of fabric softening compositions is achieved.